

Polarization and EIS studies to evaluate the effect of aluminum concentration on the corrosion behavior of SAC105 solder alloy

N. K. LIYANA¹, M. A. FAZAL^{1,2,*}, A. S. M. A. HASEEB¹

¹Department of Mechanical Engineering, University of Malaya, 50603 Kuala Lumpur, Malaysia

²Department of Mechanical Engineering, University of Jeddah, Saudi Arabia

This paper presents an investigation on corrosion behavior of Sn-1.0Ag-0.5Cu-XAl ($X = 0, 0.1, 0.5, 1.0$) by means of polarization and electrochemical impedance spectroscopy (EIS) measurements in 3.5 wt.% NaCl solution. The results show that addition of aluminum into SAC105 shifts the corrosion current density and passivation current density towards more positive values. It is also found that with an increase in aluminum concentration in SAC105 solder alloy, the corrosion current density increases and polarization resistance decreases. This suggests that SAC105 with the highest concentration of Al has the lowest corrosion resistance. In this case, the corrosion behavior seems to be attributed to anodic dissolution of aluminum and Sn-matrix.

Keywords: corrosion; lead-free solders; EIS, polarization

1. Introduction

Previously, lead solder was popularly used in electronic industries because of its satisfactory properties including good soldering, low cost, adequate melting temperature range, proper physical, mechanical, metallurgical, and fatigue resistance properties [1]. However, lead is a poisonous metal which causes huge adverse effects for human health [2]. Therefore, lead has been banned in the industry, especially in electronics field [3]. Due to health and environmental concerns induced mainly by the lead content in conventional solders, the development of lead-free solders has become an important issue for material scientists. Good wettability, low melting point, low cost, adequate strength, good corrosion resistance and thermal fatigue, etc. are the characteristics that must be fulfilled by a new lead-free solder alloy in order to replace Pb-containing solder alloys [4].

Quite a number of investigations have been done on the Pb-free solders such as binary systems Sn-Ag, Sn-Zn, Sn-Cu, etc. [5–7] and ternary

systems such as Sn-Ag-Cu, Sn-Ag-Zn, Sn-Zn-In, etc. [8]. Among these solders, ternary systems Sn-Ag-Cu with high Ag content such as Sn-4.0Ag-0.5Cu (SAC405) and Sn-3.0Ag-0.5Cu (SAC305) are considered to be promising replacements for the traditional Sn-Pb solder alloys in microelectronic applications because of their satisfactory properties [9]. However, some issues arose in these alloys, such as increase in the relative cost of the products and drop impact reliability [10–15]. Therefore, Sn-Ag-Cu alloys with low Ag content, such as Sn-1.0Ag-0.5Cu (SAC105), have been considered as a solution to these problems [13, 16, 17]. However, the thermo-mechanical fatigue properties can be affected by reducing the Ag content [18, 19]. In order to solve this problem, researchers have found a new alternative by adding fourth alloying element such as Ni, Zn, In, Al, Bi, etc. into SAC105 alloys.

Among the various choices of alloying elements, Al has received attention of researchers [20–22] because it is low cost and non-hazardous. Wei et al. [20] have investigated the effects of Al on the failure mechanism of the Sn-Ag-Zn eutectic solder. Both the ductility and tensile strength

*E-mail: fazal@uj.edu.sa

of Sn-3.7Ag-0.9Zn solder were decreased by the addition of 0.5 wt.% and 1 wt.% of Al. The effect of 0.1 wt.% and 0.2 wt.% Al on microstructure and mechanical properties of Sn-1.0Ag-0.5Cu was investigated by Dhafer et al. [21]. They reported that the addition of Al resulted in larger and less-packed intermetallic compounds (IMCs), large primary β -Sn dendrites and shrunken interdendritic regions. Lee et al. [22] investigated the effect of Al addition on the wettability and drop impact reliability of SnAgCu solder. The addition of 0.005 wt.% to 0.02 wt.% Al into SAC105 resulted in decreased wetting time and increased wetting force. However, the amount of surface oxide was increased when Al content was over 0.1 wt.%, hence, resulted in poor wetting properties.

Mohanty et al. [1] have studied the effect of Al on the electrochemical corrosion behavior of a five-element alloy. They reported that an increase in Al content decreased the corrosion current density and corrosion rate of the five-element alloy (Sn-8.5Zn-0.5Ag-XAl-0.5Ga). However, the corrosion potential value was shifted towards more noble value at higher Al content (1.5 wt.%). In another study, Mohanty et al. [4] reported that their results were opposite to the previous study. With an increase in Al content, the corrosion current density and corrosion rate of Sn-8.5Zn-XAl-0.5Ga was increased, while the corrosion potential shifted towards more negative values.

Many researchers [20, 22] have investigated the influence of Al on the microstructure and mechanical properties of the solder alloys. However, there is a lack of studies about the effect of these elements on the corrosion behavior of three-element alloys, especially SAC105. Therefore, in this study, the effect of Al concentration on the corrosion behavior of SAC105 solder alloy in 3.5 wt.% NaCl solution was investigated by polarization and electrochemical impedance spectroscopy (EIS) test.

2. Experimental

Potentiodynamic polarization test was conducted to obtain polarization curves in order to test

the corrosion resistance of solder materials using Gamry G300 Potentiostat/Galvanostat (Gamry Instruments, Inc., USA). The solders used in the corrosion tests were obtained commercially Sn-1.0Ag-0.5Cu, Sn-1.0Ag-0.5Cu-0.1Al, Sn-1.0Ag-0.5Cu-0.5Al and Sn-1.0Ag-0.5Cu-1.0Al. A 3.5 wt.% NaCl solution was used as a corrosion medium to simulate sea water. After the solder materials were cut into blocks with specific dimensions (1 cm \times 1 cm \times 1 cm), a blind hole with a 1.6 mm diameter was drilled in the blocks surfaces for electrode connection. SiC paper grits 800, 1000, 1200 and 1500 were used to polish the sample surfaces. Those were washed with distilled water to remove impurities and then cleaned with acetone.

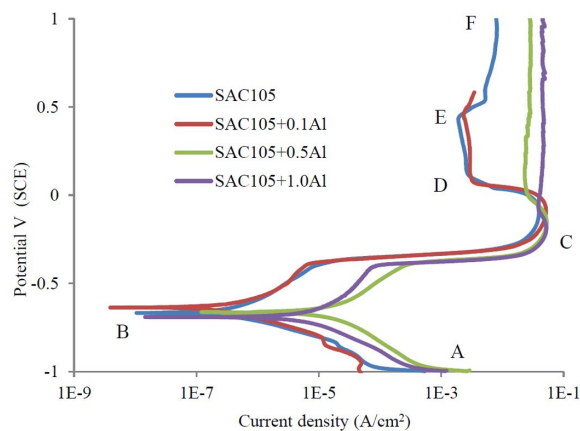


Fig. 1. Potentiodynamic polarization curves of SAC105, SAC105+0.1Al, SAC105+0.5Al and SAC105+1.0Al solder alloys in 3.5 wt.% NaCl solution.

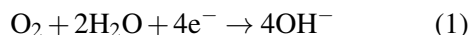
Potentiodynamic polarization studies were carried out in a cell containing 3.5 wt.% NaCl solution. A platinum wire was used as a counter electrode, and a reference electrode used in our study was a saturated calomel electrode (SCE). The solder alloy placed inside the cell served as a working electrode with the area of 1 cm². Scanning of the potential was carried out from -1 to $+1$ V at a scan rate of 1 mV s⁻¹. The EIS measurement was done at the corrosion potential in a frequency range from 100 kHz down to 0.01 Hz. After the electrochemical corrosion test, the samples

surfaces were characterized by scanning electron microscopy (SEM) equipped with energy dispersive X-ray (EDX) analysis and X-ray Diffraction (XRD).

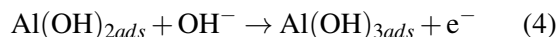
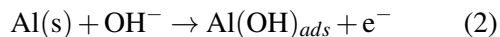
3. Results and discussion

3.1. Potentiodynamic polarization curves

The potential-current density profiles of the Sn-Ag-Cu-XAl ($X = 0, 0.1, 0.5, 1.0$) solders obtained by the potentiodynamic polarization studies carried out in 3.5 wt.% NaCl solution are presented in Fig. 1. Since all corrosion tests were performed in an aerated NaCl solution the cathodic branch of polarization curves may be ascribed to the dissolved oxygen reduction reaction:



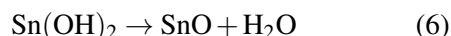
The potential at point B is referred to as E_{corr} (corrosion potential), where the extrapolated anodic and cathodic Tafel slopes intersect and the current becomes zero. Increasing the potential up to E_{corr} may induce dissolution of Al. The anodic reactions for dissolution of Al can occur by the following steps [1]:



The active dissolution of Al can be explained by the potential difference existing among the alloy elements. Table 1 shows that the electrode potential values of Ag, Cu and Sn are much higher compared with that of Al. Thus, Al has been considered as the most active among these metals. As a result, Al acts as an anode in the alloy and, therefore, more easily dissolves in corrosive conditions.

Al(OH)_3 production and deposition causes Al depletion. The active dissolution of Al continues with increasing potential until the hydroxide (OH^-) concentration reaches a critical value and supersaturates the surface of the alloy at point C.

The surface of the corroded samples covers by the insoluble hydroxide and creates a plateau region (DE) where the current density is found to be independent of potential. It should be noted that Al is the minor element in SAC105, so it is quickly depleted or blocked by Al(OH)_3 on the surface. Thus Sn, as the second most active material after Al in this system, can be dissolved to Sn(II) species [23, 24] according to the reactions displayed below:



The electrochemical data extracted from these curves are summarized in Table 2. It is seen that the SAC105 solder alloy exhibits the corrosion potential of -668 mV (SCE). With the addition of 0.1 wt.% Al, the corrosion potential increases to -637 mV (SCE). However, as 0.5 wt.% and 1.0 wt.% Al is added to the SAC105 solder alloy, the corrosion potential of the solder decreases to -669 mV (SCE) and -692 mV (SCE), respectively.

The corrosion-current density I_{corr} is determined at the intersection of the Tafel lines which represent the corrosion rate of the system. From Fig. 1, it is seen that the shapes of the polarization curves for SAC105 and SAC105 + 0.1Al solder alloys are almost similar and there is no significant difference of corrosion rate between these two alloys. The corrosion current density of SAC105 solder alloy is 0.451×10^{-3} mA/cm², which is slightly lower than that of 0.526×10^{-3} mA/cm² for SAC105 + 0.1Al solder alloy. However, the addition of 0.5 wt.% and 1.0 wt.% Al into SAC105 solder alloy shifts the corrosion current density to more positive values, which indicates an increase in corrosion rate. The corrosion current densities of SAC105 + 0.5Al and SAC105+1.0Al solder alloys are 11.1×10^{-3} and 11.6×10^{-3} mA/cm², respectively. Therefore, we can conclude that the addition of Al tends to decrease the corrosion resistance of the solder. These results are in accordance with that obtained by Mohanty et al. [1] when they investigated the effect of Al concentration on the

Table 1. Electrode potential of solder elements.

Element	Reaction	Electrode potential [V]
Silver	$\text{Ag}^{2+} + \text{e}^- = \text{Ag}$	+ 0.7996
Copper	$\text{Cu}^+ + \text{e}^- = \text{Cu}$	+0.5210
	$\text{Cu}^{2+} + 2\text{e}^- = \text{Cu}$	+0.3419
Tin	$\text{Sn}^{2+} + 2\text{e}^- = \text{Sn}$	-0.1375
Aluminum	$\text{Al}^{3+} + 3\text{e}^- = \text{Al}$	-1.6600

corrosion behavior of Sn-Zn-Al-Ga alloy. Mohanty et al. [1] reported that the corrosion current density and corrosion rate of Sn-8.5Zn-XAl-0.5Ga increased significantly with an increase in Al content. At potential more positive than B, corrosion rate increases and reaches a maximum at the passivation potential, C, which is presented by symbol, E_{pass} and critical current density defined by symbol I_{cc} . At the passivation potential, the transition from active dissolution occurs as the solid species (oxide/hydroxide of Al and Sn) become more stable than the parent metal ion [2]. The surface of the electrode is covered by these oxide/hydroxides, thereby decreasing the current density with further increment in potential to point D (Fig. 1). The current density observed in the passive region DE in Fig. 1 is referred to as the passivation current density (I_{pass}).

From Table 2, it is seen that SAC105 solder exhibits the smallest critical current density (39.8 mA/cm^2) and passivation current density (2.55 mA/cm^2). The values of current density increase as Al is added into SAC105 solder. However, the current density is found to be the lowest for 0.1 wt.% Al in comparison to other alloys containing Al, thus indicating the stability of the passive film formed on the surface. Al content of 1.0 wt.% causes an increase in the passivation current density, thereby destabilizing the passive film which results in a higher rate of corrosion of the alloy. These results are in accordance with that obtained by Mohanty et al. [1]. They reported that solder with the lowest Al content has the lowest current density.

Further increase in the potential beyond E causes a sharp increase in the current, which

indicates the transpassive region which might be due to the breakdown of the passive film by incorporation of Cl^- anion into the oxide layer, leading to localized corrosion. Previous study [25] reported that the film was mechanically broken due to the penetration of chloride ions into the oxide film and forming a solid metal chloride. The overall view from Fig. 1 shows that the addition of Al shifted the corrosion current density and passivation current density towards more positive values. This suggests that SAC105 + 1.0Al solder alloy has the lowest corrosion resistance. Higher content of Al in the Sn-1.0Ag-0.5Cu-XAl alloy enhances corrosion. These changes are also reflected in the corrosion rate as shown in Table 2.

3.2. Corrosion rate

The corrosion rate was obtained by Tafel fit using Gamry Echem Analyst DC105 software and the data are listed in Table 2. From Table 2, it is seen that SAC105 solder alloy shows the lowest corrosion rate, 0.07 mpy. When 0.1 wt.% of Al was added, the corrosion rate was slightly increased to 0.08 mpy. Corrosion rate of SAC105 solder alloy kept increasing to 1.44 mpy and 1.49 mpy when 0.5 wt.% and 1.0 wt.% Al were added, respectively. As a result, it can be concluded that SAC105 + 1.0Al solder alloy deteriorates and corrodes more rapidly with the low corrosion resistance and the highest corrosion rate.

3.3. Corrosion products

The SEM micrograph in Fig. 2a shows the formation of needle like corrosion products in SAC105 solder alloy. However, upon addition of aluminum, the size of the corrosion products seems to be smaller (Fig. 2b and Fig. 2c).

Table 2. Potentiodynamic polarization parameters for SAC105, SAC105 + 0.1Al, SAC105 + 0.5Al and SAC105 + 1.0Al solder alloys in 3.5 wt.% NaCl solution.

Materials	E_{corr} [mV]	I_{corr} [mA/cm ²]	I_{cc} [mA/cm ²]	I_{pass} [mA/cm ²]	E_{pass} [mV]	ΔE [mV]	Corrosion rate [mpy]
SAC105	−668	0.451×10^{-3}	39.8	2.55	−120.6	285.3	0.07
SAC105 + 0.1Al	−637	0.526×10^{-3}	51.3	2.98	−110.8	347.6	0.08
SAC105 + 0.5Al	−669	11.100×10^{-3}	52.1	24.30	−134.6	−	1.44
SAC105 + 1.0Al	−692	11.600×10^{-3}	52.0	38.80	−158.5	−	1.49

Samples which were potentiodynamically treated were subsequently subjected to X-ray diffraction (XRD) analysis as shown in Fig. 3. The XRD patterns reveal the presence of $\text{Sn}_3\text{O}(\text{OH})_2\text{Cl}_2$ (ICDD 00-039-0314), SnO (ICDD 98-006-019) and SnO_2 (ICDD 98-000-9178) on the exposed surface of SAC105. $\text{Al}(\text{OH})_3$ (ICDD 98-000-6162) and Al_2O_3 (ICDD 00-010-0173) were detected on the surfaces of Al-containing solder alloys.

From the XRD analysis in Fig. 3, it is seen that $\text{Sn}_3\text{O}(\text{OH})_2\text{Cl}_2$ was undetectable when Al was added into SAC105 alloy. Instead, $\text{Al}(\text{OH})_3$ and Al_2O_3 appear to be formed on the surfaces of SAC105 + 0.1Al, SAC105 + 0.5Al and SAC105 + 1.0Al solder alloys. These results are in accordance with the previous study conducted by Mohanty *et al.* [1] when they studied the effect of Al on the electrochemical corrosion behavior of Pb free Sn-8.5Zn-0.5Ag-XAl-0.5Ga solder in 3.5 wt.% NaCl solution. They reported that considerable aluminum segregation towards the surface occurred, predominantly as $\text{Al}_2\text{O}_3/\text{Al}(\text{OH})_3$, with an increase in Al content to 1.5 wt.% in the five-element solder.

3.4. Electrochemical impedance spectroscopy (EIS)

Fig. 4 displays the Nyquist plots for SAC105, SAC105 + 0.1Al, SAC105 + 0.5Al and SAC105 + 1.0Al solder alloys in 3.5 wt.% NaCl solution. As can be seen, the response of the system in the Nyquist complex plane is a semicircle arc for all materials whose diameter decreases with the addition of Al. The diameter of the Nyquist semicircle corresponds to the

corrosion resistance. It is important to remark that the highest semicircle arc corresponds to the best electrochemical corrosion behavior. In Fig. 4, it is seen that the loop diameter of SAC105 solder alloy is the highest. As Al was added into SAC105 alloy, the loop diameter decreased, which indicates the increased corrosion rate and thus, the reduction of corrosion resistance with the addition of Al. This observation permits to conclude that the SAC105 solder alloy has the best corrosion resistance as compared to others alloy.

Fig. 5 shows the impedance spectra, presented as Bode plots, for SAC105, SAC105 + 0.1Al, SAC105 + 0.5Al and SAC105 + 1.0Al solder alloys in 3.5 wt.% NaCl solution. It is well-known that the corrosion resistance of solder alloys can be determined by $\log |Z|$ (Fig. 5a) and phase angle θ_{max} (Fig. 5b) versus \log frequency. Higher $\log |Z|$ and higher θ_{max} represent better corrosion resistance. In the lower frequency region of the Bode plots (Fig. 5a), the $\log |Z|$ tends to decrease with the addition of Al and it has been tabulated in Table 3.

It can be seen from Fig. 5 that the SAC105 solder alloy exhibits the highest magnitude of impedance, $4229 \Omega \cdot \text{cm}^2$, and highest θ_{max} , -77.28° , indicating the highest corrosion resistance which is attributed to the formation of comparatively more adherent and protective oxide film on the surface. $\log |Z|$ and θ_{max} tend to decrease with the addition of Al. This demonstrates that Al can reduce the corrosion resistance of the solder. From Table 3, it is seen that the values of $\log |Z|$ and θ_{max} decrease to $3139 \Omega \cdot \text{cm}^2$ and -73.72° , respectively for SAC105 + 0.1Al and they keep to decrease to $1456 \Omega \cdot \text{cm}^2$ and -62.94° with the addition of 0.5 wt.% Al. At the highest concentration

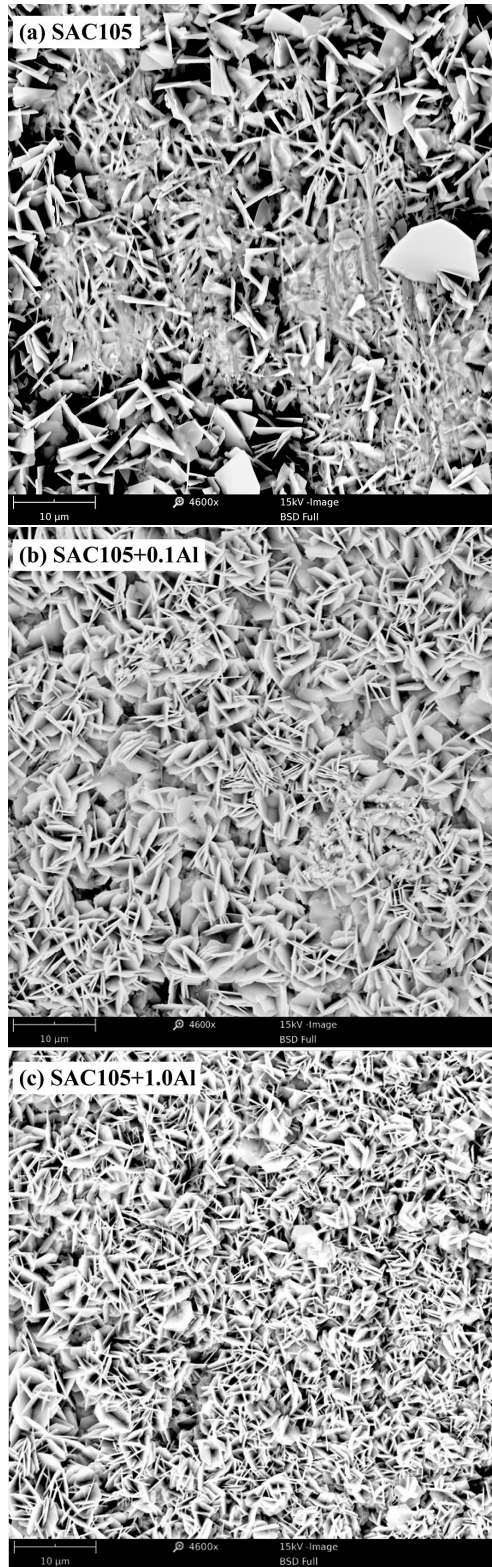


Fig. 2. SEM micrographs ($\times 4600$) of SAC105 (a), SAC105 + 0.1Al (b) and SAC105 + 1.0Al (c) solder alloys after polarization test.

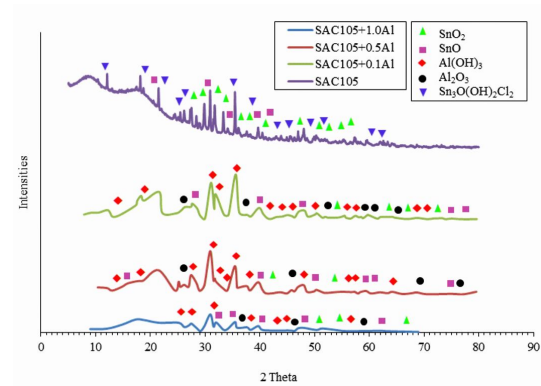


Fig. 3. X-ray diffraction scans after polarization for SAC105, SAC105 + 0.1Al, SAC105 + 0.5Al and SAC105 + 1.0Al solder alloys in 3.5 wt.% NaCl solution.

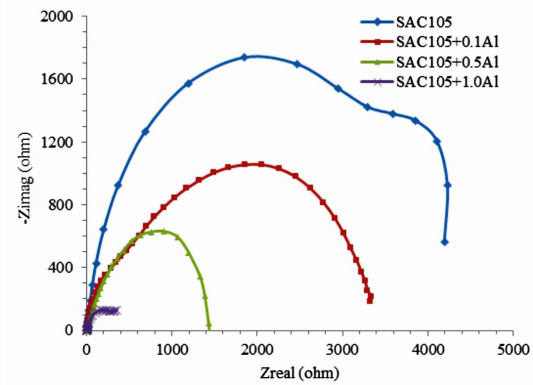


Fig. 4. Nyquist plot for SAC105, SAC105 + 0.1Al, SAC105 + 0.5Al and SAC105 + 1.0Al solder alloys in 3.5 wt.% NaCl solution.

of Al (1.0 wt.%), SAC105 alloy exhibits the lowest $\log |Z|$ ($719.7 \Omega \cdot \text{cm}^2$) and θ_{\max} (-61.89°). It can be concluded that the addition of Al into the SAC105 solder causes a decrease in the corrosion resistance of the solder and SAC105 without Al addition exhibits the best corrosion resistance as compared to the other solders.

4. Conclusions

The following conclusions could be drawn from the present study

1. The corrosion current densities of SAC105 + 0.1Al, SAC105 + 0.5Al and SAC105 + 1.0Al solder alloys

Table 3. Electrochemical impedance spectroscopy parameters for SAC105, SAC105 + 0.1Al, SAC105 + 0.5Al and SAC105 + 1.0Al solder alloys in 3.5 wt.% NaCl solution.

Solder alloys	Log $ Z $ [$\Omega \cdot \text{cm}^2$]	$\theta_{\text{max.}}$ [$^\circ$]
SAC105	4229.0	-77.28
SAC105 + 0.1Al	3139.0	-73.72
SAC105 + 0.5Al	1456.0	-62.94
SAC105 + 1.0Al	719.7	-61.89

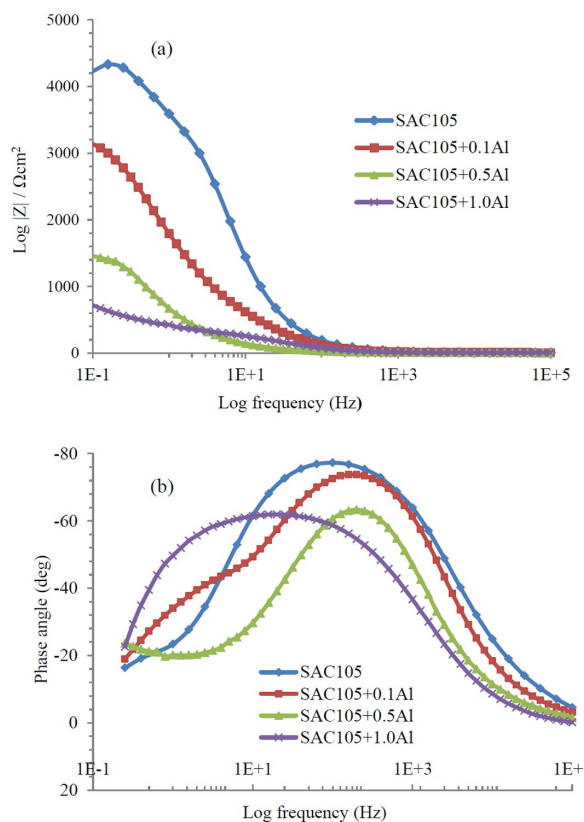


Fig. 5. 5 (a) magnitude Bode plot and (b) phase angle Bode plot for SAC105, SAC105 + 0.1Al, SAC105 + 0.5Al and SAC105 + 1.0Al solder alloys in 3.5 wt.% NaCl solution.

are 0.526×10^{-3} , 11.1×10^{-3} and 11.6×10^{-3} mA/cm², respectively. With the increase of aluminum concentration in SAC105 solder alloy, the corrosion current density increases and polarization resistance decreases. This corrosion behavior seems to be attributed to anodic dissolution of

aluminum and Sn matrix. Therefore, it can be concluded that the addition of Al decreases the corrosion resistance of the SAC105 solder alloy.

- The common corrosion products form SAC105 include SnO, SnO₂ and Sn₃O(OH)₂Cl₂. In our study, Sn₃O(OH)₂Cl₂ was undetectable when Al was added into SAC105 alloy. Instead, Al(OH)₃ and Al₂O₃ appeared to be formed on SAC105 + 0.1Al, SAC105 + 0.5Al and SAC105 + 1.0Al solder surfaces.
- Electrochemical impedance spectroscopy measurements showed that SAC105 solder alloy had the best electrochemical corrosion resistance. As Al was added into SAC105 alloy, the loop diameter was decreased which indicates the increased corrosion rate. Therefore, corrosion resistance was reduced with the addition of Al. This observation permits to conclude that the SAC105 solder alloy without Al addition has the best corrosion resistance as compared to others alloys.

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